



## **SPECIFICATION AMENDMENTS**

Please add the following paragraphs beginning on page 5, line 1, as follows:

### **DESCRIPTION OF THE INVENTION**

#### **TERMS AND LANGUAGE OF THE PROCESS**

**LAMELLAE** - Thin, plate-like structures in the micron thickness range. If present, lamellae may be viewed microscopically in microtomed cross-sections. Also referred to as fault lines.

**LAMINATIONS** - Layers of sheet composition in the mil thick range and above. Lamination may be viewed visually.

**CALENDERING (TO ORIENT)** - Passing a material between two uniform clearance even speed rolls rotating at a surface speed of approximately two feet per minute, reducing the thickness of the processed material by approximately 50 percent with each pass through the calendar at 90 degrees to the previous pass, to produce shear and working for the purpose of introducing biaxial orientation.

**CALENDERING (TO COMPACT ONLY)** - A single pass through a calender for compaction only, to laminate layers of a composite consisting of two or more layers. Shear and working of the resin is not the objective and orientation should not occur.

**ROLLING** - Results are equivalent to calendering, the choice is a matter of preference to accomplish a particular operation. Rolling is performed on a flat level surface. Accurate sheet caliper is more difficult to maintain than with calendering. Two spacers at both ends of the roll control thickness.

**Note:** When performed in identical sequence, rolling and calendering have proven to be equal.

**FILLER** - In its original context, a filler was a material added to extend the ability and reduce the cost of a polymeric material. As time passed, certain fillers were found to have functional advantages such as reducing deformation, reducing cold flow or increasing friction or improving thermal and electrical properties of the polymer.

**ADDITIVES** - Additives have usually had a special function, such as to add color, to improve adhesion, to foster nucleation and so forth.

**Note:** In essence, both fillers and additives are materials added for specific purposes. As years have passed, these two terms have, at times, been used interchangeably.

**HYDROSTATIC PRESSURE COALESCIBLE COMPOSITION** - A homogenous mixture of polytetrafluoroethylene (PTFE) colloidal resin particles, which may or may not contain submicron particulate solids up to 25 microns in size, in a liquid that wets the surface of PTFE and solids, the liquid component maintaining a volume percentage between 17 and 20 percent of the mix in compressed void free form. The condition is dependent upon the particle packing of the total solids component. Below 17 percent there is insufficient liquid to fill voids between the particles, thus promoting cavitation. Above 20 percent there is an overabundance of liquid, which promotes turbulence. In the 17 to 20 percent liquid zone, capillary forces in the spaces between packed particles are developed which draw the particles together. The resulting cohesion of particles is responsible for the surprising strength developed before a PTFE matrix is developed to further aid the development of strength.

**PASTE EXTRUSION** - Extrusion of a hydrostatic pressure coalescible composition is called paste extrusion which is performed at room temperature; the colloidal PTFE resin component has never been melted. The extrusion mold and its die components are streamlined to prevent cavitation and turbulence. This form of extrusion involves plug flow; the flow is uniaxial, or biaxial planar (also radial in special forming operations). Since the flow is of the plug type, particles all move together and no mixing occurs. For example, a tube in the extruder barrel remains a tube when extruded, but has a much smaller diameter and a thinner wall. Coagulated dispersion resin, often called fine powder, is actually a loosely aggregated particle whose average aggregate size is 500 microns, which is far from a fine powder.

**PARTICLE SIZE** - Particle size in paste extrusion is very important, but not critical to, the extrusion process if particle size remains in the recommended range, preferably up to and including 25 microns as described in this invention. Sizes above 25 microns can be employed in certain applications but rarely larger than 50 microns. Examples I and II demonstrate why PTFE particles should be colloidal and the advantage

derived in obtaining a homogenous compound in the liquid blending method. For pore forming applications, the desired fugitive particle size is generally below 10 microns and for special microfiltration applications particle sizes below 1 micron are desirable.

**MATRIX TENSILE STRENGTH** - The tensile strength based on the total cross-section, corrected for the percentage of voids in the structure employed for determining the tensile strength of porous compositions of PTFE.

**LUBRICANT** - The wetting liquid employed in paste extrusion is often described as a lubricant.

**FISH-TAIL DIE** - A stream-lined die shaped like a fish tail employed for paste extruding tape and film.

**WETTING LIQUID** - A low surface tension liquid (0 to 19 dynes/cm) that will spread on contact with a PTFE surface. Isopar H, an isoparafinic liquid often employed as a solvent but used here as a wetting and neutralizing medium to permit uninhibited mixing of particulate materials, sometimes is referred to in paste extrusion as a lubricant. Isopar H is selected here because of its inherent purity, low heat of vaporization for fast evaporation, low odor, high auto-ignition temperature and compliance with Food and Drug Administration (FDA) requirements for food and skin contact.

## **PARTICULATE MATERIALS**

### **(Additives and Fillers)**

An advantage of the present invention, as well as the co-pending application Serial Number 10/401,995) and the Roberts 3,556,161 patent lies in recognizing the inherent ability to compound, or mix, and process particulate materials in different material forms, such as solids, fibers, platelets, porous particulates, nanoparticles, and the like, with other special particulate particle forms of particulate PTFE-type resins homogeneously in a wetting liquid environment. The group of mixed particulate materials has grown over the years.

In the early years up until the 1960s, solid materials were tagged with the name "fillers" and consisted primarily of particulate carbon, graphite, bronze, chopped glass fibers and several other basic materials, employed as inexpensive extenders. In the years that followed, a much broader range of materials have been included and the term

“additives” has come into use, for example, pigments for color coding and polymers. Ever since the 1960s, both fillers and additives have been used interchangeably. Today, fillers and additives are added to provide many functional purposes and serve to improve and share the valuable properties of PTFE as a matrix for new products. For the above reasons, it is more accurate to name the solid particulate and its special function.

### **Polymeric Additives (Particle Solids)**

A. Particulate fluorocarbon resins that show adhesion to PTFE resin, 1) perfluoroalkoxy tetraethylene copolymer resin (PFA), 2) ethylenechlorotrifluoroethylene copolymer resin (E-CTFE), 3) ethylenetetrafluoroethylene copolymer resin (E-TFE), 4) poly(vinylidene fluoride) resin (PVDF), 5) tetrafluoroethylenehexafluorotripropylene copolymer resin (FEP), and 6) poly(chlorotrifluoroethylene) resin (CTFE).

B. Particulate polyether resins that show adhesion to polytetrafluoroethylene resin (PTFE), 1) polyether ether ketone resin (PEEK), 2) polyether ketone resin (PEK), and 4) polyethersulfone resin (PES).

C. Particulate polymethyl methacrylate is a fugitive resin that will decompose when heated above its melting point. In particulate form, it will mix with PTFE resin and leave voids in the PTFE matrix replicating the size of each fugitive particle.

D. Particulate polytetrafluoroethylene (PTFE) resin molding grade granular may be added beneficially up to about 50 percent of PTFE content in many compositions with colloidal particles alone or with other particulate materials. This addition will save resin cost since granular PTFE is less expensive. Particulate modified granular forms may also be added, for example “TFM”, marketed by Dyneon, for compression molding applications as a modified PTFE (1705) resin.

### **Inorganic Fillers (Particulate Solids)**

A. Particulate crystalline inorganic materials that are similar in chemical resistance to PTFE, a nitride, a diboride, silicon carbide, zirconium carbide, tungsten carbide and boroncarbide.

B. Particulate metal powders, such as gold, silver, platinum, iron, aluminum, copper, bronze, titanium and the like.

C. Particulate materials added to impart thermal and electrical conductivity,

such as carbon, graphite, silicon carbide, gold, silver and metal oxides.

D. Particulate fillers to control the friction and wear of PTFE articles, such as silicon carbide, graphite, molybdenum, chopped glass fibers and mica.

E. Particulate fillers, such as mica to improve electrical properties and carbon and graphite to conduct electricity, ceramic oxide catalysts suspended in PTFE porous membranes employed in fuel cell constructions as catalysts.

F. In some instances, for example, in making PTFE porous composition, particulate materials are added that are fugitive and can be removed by chemicals (calcium carbonate) or water, (sodium chloride).

In summary, any material capable of withstanding the fusion temperature range of PTFE (342 to 400 degrees Centigrade), may be included as long as it is useful in some way. **Caution:** do not mix explosive materials, such as thermit process components. Most importantly, in order to achieve good homogenous mixing and avoid interference with the plug flow of the paste extrusion process, it is preferable that particles be less than 25 microns in size. This size prevents cavitation and turbulence, which is detrimental to the paste extrusion process. There will be cases where particles somewhat larger than 25 microns will be an advantage, such as where the solid material added is included to improve thermal conductivity. There will also be cases where it is highly desirable to have extremely small particles, for example, in preparation of porous membrane structures and filters where the particle size will determine the pore size after the particles are extracted from the PTFE matrix.

It is equally important that the PTFE particles be small for the same reason why colloidal PTFE works and coagulated dispersion resin is a failure in the paste extrusion process where solid particulate are included. Today's art paste extrusion processes are incapable of paste extruding solid particulate particles successfully because the commercially available PTFE resin particle size averages 500 microns. A solids content of 1 to 2 percent used for pigmentation is the only useful application practiced today in the art.